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March 16, 1995

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*File + ask Art for
Comments.*

Dear Art:

Enclosed is a draft final report for subcontract XAC-13277-01. If I could receive the review comments on the report before April 20, I would appreciate it. I will be out of the country from April 27 until May 30. Thus, in order for me to provide a final final version of the report (due on or before May 23, 1995) I will have to complete it before April 27.

Sincerely,

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EFFECTS OF STORAGE ON SWITCHGRASS FOR BIOMASS-TO-ETHANOL AND
THERMOCHEMICAL FUELS PROJECT

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ABSTRACT

Sanderson, M.A., R.P. Egg, and C. Coble. 1995. Effects of storage on switchgrass for biomass-to-ethanol and thermochemical fuels project. Final report for the National Renewable Energy Laboratory for the period June 24, 1993 to March 15, 1995. Subcontract no. XAC-3-13277-01. National Renewable Energy Laboratory, Golden, CO.

We determined the effects of environmental factors on switchgrass biomass stored in large round bales as affected by protected and unprotected conditions. Additionally, we measured the losses of dry matter during the biomass harvesting process (cutting, drying, and baling) and conducted an energy analysis of the process. 'Alamo' switchgrass was harvested in November 1993 at the mature seed stage. The biomass was field dried to less than 11 to 19% moisture and baled into approximately 400-kg bales. Losses during harvesting phases were measured as differences between standing yield, windrowed yield, and by collecting biomass that was lost during the baling process. Bales were stored inside on concrete, outside on a grass sod unprotected from the elements, and outside on a gravel pad. Three bales from each treatment were destructively sampled at 4, 8, and 12 months of storage after baling. Samples of biomass were collected at cutting and baling for baseline data and samples sent to NREL. At each sampling, bales were characterized for the depth of weathered biomass, composition of weathered and unweathered biomass, and photographed. Temperature of four bales from each treatment were monitored daily for the first 28 days, then weekly until the end of the 12-month storage period. There were no differences ($P > 0.05$) among outside storage treatments in losses of biomass during the 12 months (average of 4.7%). There were no biomass losses for bales stored inside. Losses of biomass during baling ranged from 1 to 5% depending on moisture concentration in the

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biomass at baling. Larger losses were associated with drier biomass, presumably because of more shattering. Quality and quantity of runoff water from bales were not different ($P > 0.05$) from runoff water of control plots. Near infrared reflectance spectroscopy was accurate in predicting some, but not all, chemical constituents in a diverse population of biomass samples.

They reported that most weathering occurred in the surface 15 cm. Atwal et al. (1984) in Ontario, Canada reported that alfalfa (Medicago sativa L.) hay stored in large round bales and stored for six months lost 9% of the original dry matter when stored inside and 40% when stored unprotected outside on the ground. Harrigan and Rotz (1992) reported that net or twine wrapped bales stored outside in Michigan lost 16% of the original baled forage over 6 to 9 months compared with a 6% loss for large round bales stored inside. They reported that most of the losses in bales stored outside occurred in the outer 10 cm of the bales. They also noted that mostly soluble constituents of the dry matter were lost and that the fibrous portion of the biomass increased during storage. Several other studies have shown that most of the dry matter losses in large round bales during outside storage occur in the outer layer (LaFlamme, 1989; Lechtenberg et al., 1974; Bledsoe and Bales, 1992) and that techniques which reduced rain infiltration or allowed better drainage reduced storage losses (Nelson, et al., 1983; Russell and Buxton, 1985).

Agblevor et al. (1992) noted that the weathered layer (surface 10-cm crust) of sugarcane (Saccharum spp.) bagasse stored outdoors lost cell wall constituents (notably pentosans and hexosans) which reduced the yield of total hydrocarbons for pyrolysis and catalytic upgrading. The interior, however, did not change greatly. The degradation of the surface layer resulted in a 23% decrease in the total yield of hydrocarbons from bagasse piles. Wiselogle (1994) also reported significant losses of ethanol-soluble extractives in the weathered layer of switchgrass bales.

In addition to storage losses, there are unavoidable losses of dry matter during field

operations (e.g., cutting, baling, transport) and during field curing of the plant material (Rees, 1982). Rees (1982) estimated the total dry matter losses at 18 to 30% with most of the loss resulting from plant respiration during field drying. Rees' review, however, focused on haymaking [principally ryegrass (Lolium perenne)] in England. With switchgrass harvested for biomass at maturity respiratory losses likely would be low. Other studies have estimated total field harvesting losses of 20% for alfalfa (Anderson et al., 1981) and up to 40% for sweet sorghum harvested for biomass (Coble and Egg, 1987).

Biomass conversion facilities probably will have to store enough switchgrass biomass on site to bridge periods between production seasons. Storage of large quantities of biomass as large round bales could result in substantial rainfall runoff which could pose a threat to surface waters if not contained or treated. Silage effluent has been shown to have a high potential for surface water pollution because of its composition (Offer and Al-Rwidah, 1989a and 1989b; Deans and Svoboda, 1992). Runoff from unprotected large round bales would likely contain similar soluble constituents as silage effluent; however, concentrations may be lower because of the maturity of the plant material harvested for biomass. We have measured runoff amounts of up to 120 liters from single large round bales of switchgrass during rains of 5 to 8 cm at Stephenville (Sanderson, 1993, unpublished data). The runoff collected from the bales was the color of tea or coffee and likely contained many soluble biomass constituents (e.g., water soluble carbohydrates, minerals, soluble N compounds).

Detailed analysis of the chemical composition of switchgrass biomass is labor

intensive, expensive, and time consuming. Rapid methods of analysis would enable timely estimates of biomass quality, and may provide a tool for potential commercial ventures to use to grade biomass for composition as it enters the conversion plant. Near infrared reflectance spectroscopy (NIRS) has been used as a rapid analysis tool in forage research and is used commercially to estimate the lignocellulose and crude protein (nitrogen) levels in feeds and forages to balance animal rations (Barton and Burton, 1990; Barton, 1991). NIRS has been used to analyze many different substrates, even the lipid, protein, and moisture content of rainbow trout (Rasco et al., 1991). Thus, with proper calibration, NIRS should be able to analyze biomass feedstocks for lignocellulose and other organic compounds.

To paint a complete picture of harvesting and storage of switchgrass biomass, an analysis of how much energy is required for field operations and transport is necessary. Kjelgaard et al. (1981) reported that baling hay into large round bales required two manhours for 18 metric tons and consumed 68 megajoules of energy. Mowing and raking the same amount of forage required 2.6 manhours and 214 megajoules of energy, and transporting the harvested forage via wagons and movers required 3.2 manhours and 134 megajoules of energy.

The goals of the research outlined in the statement of work for this subcontract were to determine the extent of dry biomass loss during harvesting and storage of switchgrass under the ambient weather conditions of the south central U.S.

The specific objectives were:

1. To measure the loss of biomass during cutting, baling, and transport of switchgrass.
2. To characterize the dry matter losses during 12 months of storage of large round bales of switchgrass.
3. To determine the potential of rainfall runoff from switchgrass bales to become a surface water pollutant.
4. To develop a NIRS calibration set for switchgrass biomass.
5. To conduct an energy analysis of harvesting and storage of switchgrass.

METHODS

Objective 1: Field and harvest losses

Switchgrass at the Tom Mulloy farm 18 km south of Stephenville was cut and windrowed with a John Deere model 1209 mower-conditioner on 25 October 1993. The cutting width was 2.75 m. Before cutting, 10 1.3- by 4.6-m yield strips were harvested with an Almaco model FH-92 plot harvester. After cutting, five random sections (1.6-m long) were weighed in each of 10 windrows to estimate fresh windrow weight. Subsamples (500 g) from the yield strips and windrow sections were dried in cloth bags at 55°C for 48 hours to determine percent dry matter. Four 25-tiller samples were collected from random portions of the field before cutting for determining the stage of maturity and for estimating plant component composition. The height of 25 random tillers were recorded also. The standing Alamo switchgrass averaged 155 cm in height and was composed of 15.2% green leaf, 19.1% dead leaf, 10.5% sheath, and 43.3% stem. The maturity stage at cutting was 33.6, approximately the mature seed stage (according to the scale of Sanderson, 1992).

The windrows were left to dry for 5 days. The five sections in each of 10 windrows weighed on 25 October were reweighed before baling on 29 October. A John Deere model 430 large round baler was used to make bales. We began baling in the morning and had made nine large round bales by 11 a.m. These bales were weighed with a crane-mounted electronic load cell (Sensortronics model AD-4321A) and transported to the Research Center and stored inside. Nine more bales were made in the afternoon before rain forced us to quit. These bales were weighed and transported back to the Research Center and

stored inside. Thermocouples (nickel-chromium) were placed into three of the first nine bales and three of the second nine bales for temperature monitoring over the weekend (October 30 to November 1). Baling was finished (nine remaining bales were made) on the afternoon of 1 November. All bales were assigned to the proper treatments and final weights and samples taken on 1 November. Subsamples (300 to 500 g) were taken randomly from 10 windrows before baling on each day to estimate the percent moisture of the biomass at baling. In addition, a 5-kg composite sample from several windrows was taken before baling each day and sent to NREL for analysis.

To estimate biomass losses at baling, a canvas sling was attached to the undercarriage of the baler to catch material from the baling chamber (Koegel et al., 1985). In addition, plant material not picked up by the baler or not retained on the catcher was gleaned from three 3-m sections of the windrow from which a bale was made. The total length of windrow baled for each individual bale was measured so that the amount gleaned could be estimated for the whole bale. Subsamples of shattered material from the baler and that gleaned from the baled windrow were dried at 55°C for 48 hours to determine dry matter percentage. Baling losses were measured for three bales (windrows) on 29 October and five bales (windrows) on 1 November.

To estimate losses during transport, eight bales were weighed before and after loading on to a trailer for transport over the 18 km from the field to the storage site. The bales were loaded and unloaded with a bale "spear" mounted on a front end loader tractor. There was a total of 11 bales on the trailer during transport of the eight marked bales.

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Four calibrated thermocouples were inserted 18 cm deep into three bales from each treatment (the bales to be sampled at 12 months) immediately after baling. One thermocouple was inserted at the midpoint of each rounded side of the bale and at each end. Bale temperatures were measured with an electronic thermometer (Omega model HH21) and recorded daily for 28 days, then weekly thereafter until the end of the storage period. Bale temperature was taken at 8:30 a.m. each day. Ambient temperature in the building where bales were stored and at the outside storage site was measured (mercury thermometer) at the same time bale temperatures were recorded. Daily weather data (temperature, humidity, rainfall) for the length of the storage period was collected from weather station no. 36 maintained for the Texas Water Development Board at the Research and Extension Center at Stephenville. A portable weather station which automatically logged wind speed, air temperature, relative humidity, and rainfall was placed at the bale storage site also.

Objective 2: Storage Losses

We compared three treatments for effects on storage of large round bales of switchgrass: 1) bales stored unprotected outside on a grass sod, 2) bales stored inside, and 3) bales stored outside on a gravel pad.

For the inside storage treatment, bales were placed in a metal building on wooden pallets on a concrete floor. For outside storage, bales were placed on a grass sod or a gravel pad with a 1 to 2% slope. The gravel pad was 2.5-m wide and 30-cm deep. Bales

were oriented in a north-south line. The storage period was 12 months. Bales were blocked by the time of baling (morning of 29 October, afternoon of 29 October, and 1 November) and assigned to treatment-sample time combinations at random within the three blocks. The experimental design was a randomized complete block with a split-plot arrangement of treatments replicated three times. Bale storage treatments were the whole plots and sampling dates were the subplots. Each bale was an experimental unit.

Bales were weighed and destructively sampled at 4, 8, and 12 months after baling. Three bales from each treatment were destructively sampled at each date. At each destructive sampling, three bales from each treatment were sectioned with a chain saw to obtain a biomass sample from the unweathered core and a sample of the visibly weathered outside layer. Approximately 50 to 75 cm of biomass was removed from each end of the bale to expose the unweathered inner core and the visibly weathered layer. The thickness of the visibly weathered layer was measured at the 12, 3, 6, and 9 o'clock positions on the cut bale face of each rounded end. A 5-kg sample of the weathered layer and the unweathered core was obtained and packaged and sent to NREL via overnight express at each sampling date. A similar sample was retained at Stephenville. A 300- to 500-g subsample of the weathered and nonweathered layer along with a subsample of the bale bottom was taken to estimate dry matter percentage to adjust bale weights on an oven-dry basis. Subsamples were dried in cloth bags at 55 °C in a forced draft oven for 48 hours to determine dry matter percentage. Sampling and data collection procedures, condition of the bales, and bale environment at each date were documented via photographic slides and

videotape.

Objective 3: Runoff water quantity and quality

Runoff water amount and quality was measured by placing bales in runoff plots. The plots were 2-m wide and 6-m long. Metal flashing was installed around the perimeter of the plots to a depth of 10 cm with 10 cm of flashing above ground. The flashing directed the runoff into a covered collection tank at the base of the plot. Nine runoff plots were constructed. Large round bales of switchgrass (6-ft diameter by 4-ft long, approximately 800 lb) were placed in three of the plots, three plots contained a similar sized bale sealed in plastic, and three plots contained no bales. A bermudagrass sod was maintained at a 6-cm height in the plots that contained no bales. The experimental design was completely randomized with three replicates (plots) per treatment. Rain and runoff water amounts were measured and collected after each rainfall during 1 November 1993 to 1 November 1994. Rainfall was collected in two 1-l plastic beakers for comparative analysis. Samples were stored frozen for later analysis. All rainfall and runoff water samples were analyzed in duplicate (if enough sample was available) for total Kjeldahl N (TKN), chemical oxygen demand (COD), total solids (TS), volatile solids (VS), fixed solids (FS), total dissolved solids (TSS), volatile dissolved solids (VSS), and fixed dissolved solids (FSS) (APHA, 1992) at the Wastewater Laboratory in the Agricultural Engineering Department at Texas A&M University. Definitions of water quality terms are in Appendix A.

Objective 4: NIRS Calibration:

Coarsely ground samples (121 total) of switchgrass (56), bagasse (4), corn stover (Zea mays L.) (13), lespedeza (Lespedeza cuneata) (18), and various woody species (30) were received from Foster Agblevor of NREL for this objective. The samples were ground in a cyclone mill to pass a 1-mm screen then scanned with near infrared radiation in a Pacific Scientific Model 6250 scanning monochromator near infrared reflectance spectrometer. The samples were scanned four times on two separate days. Back-to-back duplicate scans were done on separate days. The four scans were averaged for each sample. During each scan, the sample was rotated in a spinning cup and irradiated with near infrared radiation of 1100 to 2500 nm wavelength. Each individual scan was the average of 64 scans over the NIR wavelength range. Data were recorded as $\log(1/\text{reflectance})$ at 2-nm increments for a total of 700 data points for each scan. Before scanning biomass samples each day, a reference sample was scanned to assure that the instrument was operating properly.

Twenty samples were selected randomly from the entire sample set to be used for an independent validation of the prediction equations. The remaining 101 samples were used to construct a calibration equation for predicting the concentrations of extractives, ash, lignin, uronic sugars, arabinose, xylose, mannose, galactose, glucose, total sugars, C, H, O, N, and S. Wet chemistry analytical values provided by NREL for each sample were used to relate spectral data to chemical composition for calibration. A modified partial least squares statistical technique (Infrasoft International, Port Matilda, PA; Shenk and

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Westerhaus, 1991) was used to develop the calibration equations. Calibrations were computed using log 1/R data which was scatter-corrected with the standard normal variate-detrend (SNV-D) procedure of the software. A noise repeatability file was included to help correct for daily variations in the laboratory environment and sample handling conditions.

The calibration equations were tested for predictive ability by applying them to the 20 samples reserved for independent validation. As another independent test of the prediction equations, National Institute of Standards (NIST) samples 8491 (bagasse, Saccharum spp. hybrid), 8492 (Populus deltoides), 8493 (Pinus radiata), and 8494 [wheat straw (Triticum spp.)] were scanned four times and chemical composition predicted with the calibration equations.

All NIRS scanning, calibration, and validation procedures followed recommendations of Windham et al. (1989).

Objective 5: Energy Analysis

In this study we analyzed the energy requirement for the harvesting and storage of switchgrass. The study analyzed the typical type and size of harvesting and transport equipment that would be used in an operation of this nature based of similar analyses for hay production. The analyses were used to develop a spreadsheet model so that inputs could be changed as desired. The operations analyzed included cutting, raking, baling, and transport to a central processing plant. Energy (fuel consumption) requirements were calculated for each operation for 50 tons of hay. The energy requirement per unit weight of

hay was then calculated.

Analysis of Biomass Samples

Biomass samples collected for Objectives 1, 2, and 3 were dried at 55 °C for 48 hours, ground in a Wiley mill to pass a 2-mm screen, then through a cyclone mill to pass a 1-mm screen and stored at room temperature in Nalgene bottles. Samples were analyzed for total Kjeldahl N (AOAC, 1990).

Quality Assurance-Quality Control

The main QA/QC activities were balance calibration, data archiving, sample archiving and identification, and water quality analysis results.

Laboratory balances were zeroed and calibrated before use with a 10-g and 500-g weight. The load cell used to weigh the large round bales was zeroed before each bale weight, and also checked with 48-kg and 230-kg reference weights before weighing bales. All scales remained in calibration during the study.

Samples collected from each bale sampling and each runoff event were recorded on a tally sheet for inventory purposes. An archive sample of all water and biomass samples was retained at the Stephenville Research Center.

All data on computer were stored in four forms: (1) the original raw data sheets were kept in the data notebook in the project manager's office; (2) each data sheet was photocopied and copies collected weekly and stored in a separate building; (3) data entered on computer disk were backed up on two separate disks with one disk stored in a separate

building; and (4) printouts of each file were collected weekly and stored in a separate building. All floppy disks are checked for the presence of viruses.

In the water quality laboratory at Texas A&M University, water samples were assigned a laboratory number and all analyses were done in duplicate (if enough sample was available) with the exception of nitrogen. Only one nitrogen analysis was done per sample because of the large volume of sample required (500 ml) and the low levels of N that were detected. Results were recorded on a sample results form and then manually entered into a spreadsheet where duplicates are averaged and relative standard deviations (RSD) were calculated (examples of the results form and spreadsheet are attached). In general, for RSDs 20% or greater the analysis was redone if enough sample remained. Sometimes the limited amount of runoff water precluded reanalysis or even duplicate analysis. An exception to the 20% rule was when chemical oxygen demand (COD) levels fell below 100 mg/l. At these very low levels very small differences in titrant amounts (e.g., 1 drop) can result in a very large RSD.

All thermocouples were calibrated before inserting in the bales at the start of the experiment and after removing them at the end. The thermocouples were calibrated in boiling water (100°C) and ice water (0°C). All thermocouples were within +/- 1.5°C of actual temperature. One thermocouple failed during the experiment and was replaced.

Water samples were collected as soon as practical after rainfall, labelled, recorded on an inventory sheet, then frozen for storage until analysis. An archive subsample of each water sample was retained at Stephenville.

All TKN analyses were done in duplicate. When duplicate analyses did not agree to within 5%, the sample was rerun. National Institute of Standards sample 1547 (peach leaves) was used as a reference check for the method. Given TKN for the sample was 2.94 % \pm 0.12%. Our results were 2.92% \pm 0.08% (n=13).

RESULTS

Objective 1 The average biomass loss during baling was 3.38% for the eight bales (Table 1). Baling losses were higher (4.34%) for windrows 6 to 10 which were baled on 11/1/93 when the biomass was 11% moisture than when windrows 1 to 3 were bales (1.77% loss) on 19 October 1993 when biomass was 19.8% moisture. Windrows 4 and 5 were not measured because of rain on 29 October 1993. Bale weight changes and biomass loss during handling and transport over 11 miles were very small (Table 2).

On 23 November 1994, we measured the losses of biomass during baling of Alamo switchgrass at the Stephenville Research Center. Shatter losses were collected during the making of four bales and material left behind by the baler was gleaned from the stubble for each bale. Total biomass yield from the 0.4-ha area was 9980 kg of dry biomass per ha. Percent moisture of the biomass at baling was 17.2%. The losses from baling the four bales were 25 kg/ha or 0.25% of the standing biomass. The losses from biomass remaining on the stubble after baling were 535 kg/ha or 5.4% of the standing biomass. Thus, the total loss during baling was 5.65% of the standing biomass. The large amount of biomass left behind by the baler was probably due to the large windrows resulting from the relatively high yields per acre. In November 1993, we estimated that losses from baling averaged 1.77% for 19% moisture biomass and 4.34% for 10% moisture biomass with shatter losses accounting for most of the loss. Biomass yield at that time was 6302 kg/ha with correspondingly smaller windrows than were made in 1994. These losses could be reduced by careful machine operation and management.

Table 1. Estimates of biomass losses during baling in November 1993.

Windrow	Amount gleaned	Amount collected at baler	Total loss/bale	Dry bale weight	% loss
	kg				
1	4.08	0.38	4.46	377.4	1.18
2	4.52	0.48	5.00	380.4	1.31
3	10.25	0.22	10.47	371.0	2.82
6	19.49	1.81	21.30	375.8	5.67
7	15.68	2.42	18.10	368.8	4.91
8	11.25	2.34	13.59	372.0	3.65
9	8.19	1.72	9.91	354.3	2.80
10	15.58	1.45	17.03	365.0	4.66

Table 2. Weight losses of individual bales during transport in November 1993.

	Bale weight (kg)			
Bale	Before transport	After transport	Difference (kg)	% change
14.1	474	472	2	0.42
15	498	496	2	0.40
16	468	464	4	0.85
17	492	487	5	1.02
18	497	493	4	0.80
26	450	450	0	0
9	400	400	0	0
10	406	406	0	0

Objective 2 Biomass losses in bales stored outside either on sod or gravel were 5.5 and 3.9% of the original bale weight, respectively, and were not significantly different (Table 3). No weight losses were detected in the bales stored inside. The bales stored inside had no visibly weathered layer, whereas the bales stored outside had a visibly weathered layer that slowly increased in depth during the storage period. There were no significant differences between the outside storage treatments in depth of the weathered layer.

Temperatures of the bales during storage are presented in Figures 1, 2, and 3.

Temperatures at the beginning of the storage period remained below 20 °C (except for one date) and more or less varied with the ambient temperature. There did not appear to be any heating due to microbial respiration or spoilage.

Although there was no difference between outside storage treatments in biomass loss or weathered layer, bales that were stored on the ground had a large, black, rotted area where the biomass was in contact with the ground. The bales stored on gravel did not have this area of spoilage. In fact, the biomass on the bottom of the bale in contact with the gravel remained green and dry. Thus, even though the losses in biomass were not different, an inexpensive gravel storage area may be worthwhile to prevent the deterioration of the bottom of bales and to allow drainage of water away from the bales. Johnson (1993) reported losses of 8 to 15% for twine-tied switchgrass bales stored on sod compared to losses of 3.4% for bales stored on crushed rock in Indiana. Biomass losses in our study were lower than the 13 to 19% losses we reported in previous studies (Sanderson et al., 1992; Sanderson and Ward, 1994). This may be partly attributed to differences in bale

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sizes between the experiments. Bales in the earlier studies were 1.38-m in diameter and weighed approximately 275 kg, whereas bales in the present study were 1.75-m in diameter and weighed approximately 410 kg. Buckmaster (1993) indicated that spoilage losses in large round hay bales increased as bale diameter decreased.

Nitrogen concentration was not affected ($P < 0.05$) by the windrowing or baling operations (Table 4); however, N concentrations in the shattered biomass collected from the baler was slightly higher in N probably because leaves, which typically are higher in N, made up most of the shattered material.

Nitrogen concentration in material from the bottom of sod-stored bales was higher ($P < 0.05$) than at other positions (Table 5). Nitrogen concentrations did not differ ($P > 0.05$) among positions for the inside or gravel storage treatments. This may have been caused by leaching of other nutrients resulting in increased fiber concentrations in rotted material from the bottom of the bale. Most of the nitrogen in grasses that are very mature is associated with fiber or cell walls and is not soluble.

Table 3. Dry weights of bales immediately after baling, and at 4, 8, and 12 months of storage in 1993 to 1994. Data are means of three replicate bales per date.

Treatment	At baling	4 months	8 months	12 months	kg loss	% loss
bale weight (kg)						
Inside storage	379	399	389	385	0	0
Outside storage-sod	396	377	370	375	21.8	5.5
Outside storage-gravel	378	383	375	363	14.6	3.9
		Depth of weathered layer (cm)				
Inside storage	0	0	0	0		
Outside storage-sod	0	9.9	12.4	13.3		
Outside storage-gravel	0	8.4	11.2	12.3		

Table 4. Concentrations of total Kjeldahl nitrogen at various times during harvest of switchgrass biomass in November 1993.

Date	Source	% N	SD	n
10/25/93	Standing crop	0.47	0.07	10
10/25/93	Windrow sample	0.55	0.08	10
10/29/93	Windrow sample	0.51	0.10	10
11/1/93	Windrow sample	0.48	0.06	5
10/29/93	Bale shatter	0.63	0.06	10
10/29/93 and 11/1/93	Windrow gleanings	0.48	0.05	8

Table 5. Concentration of total Kjeldahl nitrogen in various portions of switchgrass bales during storage for one year.

		Sampling date			
Bale storage treatment	Bale portion	March 1	July 1	November 1	Average
Total Kjeldahl N (% of dry matter)					
Sod	Exterior	0.43	0.50	0.50	0.48
	Interior	0.44	0.44	0.44	0.44
	Bottom	0.51	0.69	0.50	0.57
	Average	0.46	0.54	0.48	0.50
Inside	Exterior	0.52	0.53	0.41	0.49
	Interior	0.47	0.45	0.45	0.46
	Bottom	0.51	0.50	0.46	0.49
	Average	0.50	0.49	0.44	0.48
Gravel	Exterior	0.50	0.50	0.49	0.50
	Interior	0.45	0.51	0.48	0.48
	Bottom	0.47	0.50	0.47	0.48
	Average	0.47	0.50	0.48	0.48

Objective 3 Runoff amounts collected from each treatment along with rainfall at each runoff event are presented in Figure 4. Runoff was collected from each treatment at 27 different dates from November 1993 to October 1994. Runoff amounts varied from less than one liter to over 90 liters depending on rainfall amounts. As expected, runoff increased with increased rainfall amount and intensity. There were no significant

differences among treatments in runoff volume.

Although runoff was collected at 27 dates, some treatments and replicates did not produce enough runoff for analysis. There were only 12 dates for which there were complete water quality data for all replicates of each treatment. An analysis of variance was conducted on data from each complete date. There were significant differences among treatments for only one or two constituents on three dates.

The average concentrations of water quality constituents for all 12 dates are in Table 6. Levels of all constituents were low and would probably not pose a significant threat to contamination of surface water. Concentrations of all constituents in runoff water peaked during December 1994. The high solids concentration at this time indicates that soil from the edges of the flashing in the runoff plots or from around the bottom of the bales may have washed away and into the collection tanks. Runoff amounts during this time were low corresponding to the low rainfall, and any soil particles in the small sample may have elevated the concentrations of several constituents.

Fifty to 60% of the TS in each treatment was present as FS, which is an estimate of the inorganic components (Metcalf and Eddy, Inc., 1991), in this case, an indicator of the presence of soil particles (Table 6). Volatile solids are an estimate of the organic matter present in the water. Of the VDS, roughly 50% was present as fixed solids. Compared with rainwater, runoff water from all treatments was enriched in each water quality constituent. John M. Sweeten (Dep. of Agric. Eng., Texas A&M University, personal communication) collected rainwater samples near Stephenville and found concentrations of

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16 mg l⁻¹ COD, 80 mg l⁻¹ TS, 56 mg l⁻¹ FS, 24 mg l⁻¹ VS, 20 mg l⁻¹ TDS, 0.22 mg l⁻¹ TKN, and 0.63 mg l⁻¹ NH₃-N, very similar to our results for rainwater.

The concentrations of water quality constituents in runoff water from our experiment were similar to those reported by others for runoff from a grass sod. Edwards and Daniels (1993) reported concentrations of 5.2 mg l⁻¹ TKN, 1.6 mg l⁻¹ NH₃-N, 80 mg l⁻¹ COD, and 7.2 mg l⁻¹ of TSS in runoff from tall fescue (*Festuca arundinaceae* Schreb.) generated with a rainfall simulator. Similar plots treated with swine manure had concentrations of 58 mg l⁻¹ TKN, 40 mg l⁻¹ NH₃-N, 410 mg l⁻¹ COD, and 79 mg l⁻¹ TSS. In a study conducted by the Texas A&M Research and Extension Center at Stephenville and the Agric. Eng. Dep. at Texas A&M University, we found concentrations of 39 mg l⁻¹ COD, 476 mg l⁻¹ TS, 392 mg l⁻¹ FS, 84 mg l⁻¹ VS, 511 mg l⁻¹ TDS, 8.6 mg l⁻¹ TKN, and 0.31 mg l⁻¹ NH₃-N in runoff water from large (40-ft by 60-ft) plots of bermudagrass near Stephenville. As an extreme example, Sweeten and Wolfe (1994) reported average concentrations of 2088 mg l⁻¹ TS, 966 mg l⁻¹ VS, 1480 mg l⁻¹ COD, 172 mg l⁻¹ TKN, and 161 mg l⁻¹ NH₃-N in samples of effluent from several dairies near Stephenville. Values for water quality constituents in runoff water from several agricultural operations are in Table 7.

We conclude that runoff water from large round bales of switchgrass does not differ in water quality from that of runoff water from grass sod. Concentrations of water quality constituents were low and should not pose a hazard to contamination of surface water and should not present a problem for municipal or industrial stormwater systems. It should be noted, however, that we used a small system--one bale. We do not know if runoff water

from large stockpiles of bales react similarly.

Table 6. Concentrations of water quality constituents in runoff water from three treatments. Data are averages of three replicates per treatment and 12 collection dates.

	Treatment			
Item	Sod control	Plastic control	Bales	Rainwater
	mg l ⁻¹			
Chemical oxygen demand	152	119	163	26
Total solids	248	217	323	64
Fixed solids	125	132	200	53
Volatile solids	113	85	126	23
Total dissolved solids	179	171	213	59
Volatile dissolved solids	81	68	97	20
Fixed dissolved solids	91	102	107	57
NH ₃ -N	2.68	2.02	3.31	0.40
TKN	3.94	2.81	4.76	0.50

Table 7. Concentrations of water quality constituents from a variety of runoff sources and agricultural operations.

Source	Concentration (mg/l)						Ref
	COD	TKN	NH ₄ -N	TS	FS	VS	
Beef feedlot (dirt)	17800–2160	17500–3000		2986–17500			1
Range or pasture (well managed)		0.5–2.5					2
Range or pasture (poorly managed)		3–15					2
Beef feedlot (dirt)	2900–28000	9–280	2–85				3
Beef feedlot (concrete)	8400–32800	70–1070	33–775				3
Precipitation	9–16	1.2–1.3					3
Forested land		0.3–1.8					3
Crop land	80	9					3
Dairy yard runoff	312–1504	22.1–154.0	7.3–75.5	624–3538			4
Beef feedlot runoff holding ponds	1100	180		2470			5
Beef feedlot runoff holding ponds	184–11784	12–396	12–345	610–28543		130–760	6 3
Dairy wastewater milking parlor wastewater	6397	260	248	5541	2096	3444	7
Dairy wastewater two-stage lagoon effluent	650	117	116	1644	963	681	7
Dairy wastewater settling basin outflow	6086	305	305	5127	2016	3111	7

Dairy wastewater primary lagoon effluent	5467	282	267	5068	2069	2999	7
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This Study

Blank	225.4	5.2	3.1	391	240	171
Plastic	195.5	3.9	2.8	361	216	160
Bale	207.5	4.4	3.1	361	210	153
Rain	26.0	0.5	0.4	64	53	23

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Objective 4 Spectra of representative samples of Alamo switchgrass, black locust, bagasse, lespedeza, corn stover, and hybrid poplar are in Figure 6. The major differences among samples appeared to be at the region of 1200 to 1500 nm and 2100 to 2500 nm.

Calibration and validation statistics are in Tables 8 and 9. The SEC describes how well the calibration samples were fit. The lower the SEC the better the fit. Calibration errors (SEC), as a percentage of the mean, were largest for extractives, uronic sugars, mannose, and sulfur. Very low SEC values were obtained for lignin, arabinose, xylose, glucose, and total sugars. The R^2 values were high for all constituents except hydrogen, oxygen, and sulfur. In general, the prediction equations did a fair job of predicting most constituents. The prediction equations worked best for lignin, arabinose, and nitrogen. The accuracy of the equations for extractives, ash, xylose, glucose, carbon, and total sugars was reasonable, but there was some unacceptable variation. The prediction equations were not accurate for uronic sugars, mannose, galactose, oxygen, hydrogen, or sulfur. These constituents were present in very low amounts or the range in variability was very limited, which causes difficulty in NIRS calibration.

As an independent test of the prediction equations, National Institute of Standards and Technology (NIST) samples 8491 (bagasse), 8492 (Populus deltoides), 8493 (Pinus radiata), and 8484 (wheat straw) were scanned four times and chemical composition predicted with the calibration equations. Results for individual scans and the actual values given by NREL are in Table 10. Pinus radiata and wheat straw samples were not included in the original calibration set, thus, inaccurate results for some constituents predicted for these NIST

samples are not surprising.

The SEP is a true indication of the performance of the equations on unknowns from the same population. The values of bias, SEP(C), or r^2 that are marked by superscripts indicate that these values exceeded the control limits of acceptable equation performance. The control limits are (1) SEP(C) cannot exceed $1.3 \cdot \text{SEC}$; (2) the absolute value of bias cannot exceed $0.6 \cdot \text{SEC}$; and (3) the r^2 should be above at least 0.80. For most constituents that exceeded the control limits, the SEP(C) was greater than $1.3 \cdot \text{SEC}$. This was caused primarily by one or two samples, common to all constituents, which could not be predicted accurately. These most often were sycamore, eucalyptus, or a corn stover sample. There were very few sycamore or eucalyptus samples in the calibration set, thus, it is not surprising that they could not be predicted.

Plots of predicted values versus observed values for the 20 independent samples reserved for validation are in Figures 7, 8, and 9. Plots of C, H, O, and S are not presented because the prediction accuracy was very poor. In many instances, the predicted values were nearly the same as the observed values, especially for lignin and arabinose (Fig. 7). There was a great deal of variation about the regression line for several of the structural carbohydrates (Fig. 8). The calibration equation predicted negative values for nitrogen in eucalyptus biomass and these values were eliminated from the validation set. Eucalyptus samples, however, were very low in N (0.05% N).

These data indicate that NIRS can be used to predict the chemical composition of a broad range of biomass feedstocks. What is remarkable is that given the good calibration and

validation results for some constituents of such a diverse population, it appears that one broad-based equation could be used for many feedstocks, thereby reducing calibration costs. It is recommended that NREL maintain a large library of diverse feedstock samples along with a database of accurate and precise chemical composition data, for future commercial development of NIRS analysis of biomass feedstocks.

Table 8. Calibration statistics for 101 samples of various biomass feedstocks.

Constituent	n	Mean	SEC†	R ²	SECV
Extractives	96‡	8.58	1.10	0.96	1.31
Ash	96	3.86	0.36	0.97	0.44
Lignin	97	21.73	0.73	0.97	0.81
Uronic sugars	71	3.07	0.49	0.89	0.57
Arabinose	99	2.09	0.11	0.99	0.13
Xylose	95	18.90	0.65	0.97	0.77
Mannose	96	1.05	0.12	0.98	0.16
Galactose	95	1.05	0.08	0.94	0.10
Glucose	99	36.87	1.05	0.95	1.34
Total sugars	98	59.93	1.24	0.92	1.56
Carbon	75	47.77	0.37	0.93	0.43
Hydrogen	75	5.85	0.21	0.23	0.22
Nitrogen	81	0.69	0.06	0.97	0.07
Oxygen	45	40.83	0.87	0.68	0.99
Sulfur	49	0.06	0.01	0.82	0.02

†SEC = standard error of calibration; R² = squared coefficient of multiple determination from the modified partial least squares regression of known values on NIRS values; SECV = standard error of cross validation with four groups.

‡n is less than 101 for all constituents because of incomplete data for some samples and the elimination of outliers during the calibration process.

The SEC describes how well the calibration samples were fit. The lower the SEC the better the fit.

R² is the proportion of variation explained by the regression equation.

Table 9. Prediction statistics of calibrations tested on 20 independent biomass feedstock samples.

Constituent	n	Mean	SEP†	Bias	SEP(C)	r ²
Extractives	20	7.88	1.10	0.18	1.11	0.96
Ash	20	3.64	0.60	-0.21	0.58‡	0.93
Lignin	20	22.22	0.70	-0.32	0.64	0.98
Uronic sugars	16	3.21	0.73	0.12	0.74	0.76
Arabinose	20	2.05	0.12	0.01	0.12	0.99
Xylose	20	18.88	1.17	-0.09	1.20‡	0.92
Mannose	20	0.99	0.32	-0.06	0.32‡	0.88
Galactose	20	1.06	0.12	0	0.12‡	0.87
Glucose	20	37.44	1.45	0.66§	1.32	0.93
Total sugars	20	60.41	1.80	0.60	1.74‡	0.83
Carbon	17	47.89	0.91	-0.25§	0.90‡	0.73¶
Hydrogen	17	5.75	0.22	-0.06	0.22	0.12¶
Nitrogen	18	0.60	0.10	-0.02	0.10‡	0.93
Oxygen	12	40.55	1.23	-0.13	1.28‡	0.65¶
Sulfur	12	0.05	0.02	0	0.02‡	0.68¶

†SEP = standard error of performance; bias = difference between predicted and actual mean for validation set; SEP(C) = standard error of performance corrected for bias; r² = squared coefficient of simple determination from regression of known values on NIRS predicted values.

‡SEP(C) value is > 1.3*SEC.

§Absolute value of bias is > 0.6*SEC.

¶r² is unacceptably low.

n is less than 20 for some constituents because of incomplete data for some samples.

Table 10. NIR analysis of NIST samples compared with actual values.

Sample	Scan	Extractives	Ash	Lignin	Uronic sugars	Arabinose	Xylose	Mannose	Galactose	Glucose	Total sugars	C	N
<u>Pinus radiata</u>	1	2.24	-0.77	28.56	5.13	0.27	13.29	3.48	0.94	51.37	67.11	49.48	0
	2	1.93	-0.79	28.64	5.13	0.24	13.53	3.54	0.92	51.60	67.58	49.52	0
	3	2.05	-0.79	28.74	5.06	0.29	13.58	3.49	0.94	51.45	67.47	49.51	0
	4	1.95	-0.78	28.64	5.14	0.25	13.57	3.53	0.93	51.55	67.55	49.52	0
	Avg.	2.04	-0.78	28.64	5.11	0.26	13.49	3.51	0.93	51.49	67.42	49.51	0
	Actual	2.70	0.30	25.90	2.50	1.50	10.70	5.90	2.40	41.70	62.20	50.26	0.03
Bagasse	1	2.77	3.32	24.46	2.12	2.00	22.49	0.08	0.65	40.03	65.57	48.52	0.13
	2	2.72	3.34	24.55	2.05	2.00	22.54	0.09	0.65	40.21	65.80	48.55	0.13
	3	3.12	3.09	24.14	2.04	1.99	22.64	0.04	0.62	39.85	65.62	48.50	0.12
	4	2.95	3.02	24.43	2.12	1.97	22.84	0.10	0.64	39.83	65.89	48.55	0.12
	Avg.	2.89	3.19	24.40	2.08	1.99	22.63	0.08	0.64	39.98	65.72	48.53	0.12
	Actual	4.40	4.00	23.10	1.20	1.70	20.40	0.30	0.60	38.60	61.60	47.57	0.17
Wheat straw	1	13.86	6.43	17.49	3.01	2.75	23.08	0.23	1.03	33.67	56.69	46.25	0.72
	2	13.74	6.37	17.67	3.02	2.72	22.99	0.28	1.04	33.83	56.82	46.30	0.72
	3	14.14	6.33	17.45	3.00	2.74	23.09	0.22	1.02	33.38	56.53	46.28	0.72
	4	14.02	6.29	17.60	3.00	2.75	23.22	0.25	1.03	33.43	56.75	46.30	0.71
	Avg.	13.94	6.36	17.55	3.00	2.74	23.10	0.24	1.03	33.58	56.70	46.28	0.72
	Actual	12.95	10.22	16.85	2.24	2.35	19.22	0.31	0.75	32.64	55.27	43.88	0.63

Objective 5. The main energy requiring components of harvesting switchgrass for biomass utilization include cutting, raking baling, transport to roadside, and transport to a central location. The equipment needed during the harvest process includes a tractor, mower conditioner, hay rake, round baler that produces large bales, three-point hitch mounted bale mover, and a truck-trailer transport setup. Because the power required to form large round bales may exceed 50 hp, a tractor with a power rating in the range of 70 – 80 hp was chosen to provide sufficient power throughout the process (Freeland et al., 1988). A tractor this size typically weighs approximately 9,000 lbs and has a front-to-rear weight distribution of 1.3. A rough estimate of the fuel efficiency of a 70 – 80 hp tractor is approximately 13.0 hp-hr per gallon of diesel (Wendel, 1985). The specific energy consumption of a forage harvester has been estimated to range between 5 and 8 MJ per tonne of wet matter (Savoie et al., 1987). For normal operations, an average value of 6.5 MJ per tonne of wet matter was used. The total energy consumption of the forage cutting operation was calculated to be approximately 5,600 Btu per ton of hay.

The energy requirement for raking the hay with a side delivery rake was calculated based on the energy required to overcome the motion resistance of the tractor and rake as they move across a field. An average rake weighs approximately 500 lb and will travel approximately 1000 ft per ton of hay. The energy consumption of the hay raking was calculated to be approximately 3,600 Btu per ton of hay. The energy required at the hay baler to form large round hay bales is approximately 5,100 Btu per ton of hay (Freeland et al., 1988). Based on DRAFT FINAL REPORT, Sanderson, Egg, Coble. NREL subcontract XAC-3-13277-01.

the fuel efficiency of the tractor, the total amount of energy required is approximately 21,000 Btu per ton of hay. After the hay has been baled, each bale must be transported to a load-out area where it is loaded on a truck for shipping. The energy required for this operation can be estimated by the energy required to overcome the motion resistance of the tractor and hay bale. The same tractor dimensions and methods used for raking was used. It was assumed that each bale must be moved 1/3 mile giving an energy requirement of 13,000 Btu per ton of hay. There are two methods for shipping the large round hay bales: (1) a tractor-trailer which can carry approximately 20 bales per load or (2) a pickup truck-trailer that can carry approximately 6 bales per load. The easiest method for calculating the energy requirements for each method is based on specific fuel consumption. The tractor-trailer has a specific fuel consumption of approximately 6.2 miles per gallon. The specific fuel consumption for the pick-up truck-trailer system is approximately 10 miles per gallon (Desfor et al., 1975). The mean round-trip distance between farm and a processing plant was assumed to be 20 miles. For the tractor trailer, the energy requirement was calculated to be 45,000 Btu per ton of hay. The pickup truck-trailer had an energy requirement of approximately 92,000 Btu per ton of hay. These values were used in a spreadsheet model (Table 11) that calculated the fuel and energy consumption of each of the operations for 50 tons of hay. The spread sheet also calculated the energy requirement and output fuel energy (alcohol): harvest/transport energy per ton of hay. The energy required for harvest transport was found to be 105,500 Btu/ton of hay and the output fuel energy (alcohol): harvest/transport energy per ton of hay was 55.4. The harvesting/transport energy was only a small fraction of the energy potential of the hay.

Table 11. Harvesting and Transport Energy Requirements for Switchgrass Hay.

Inputs and Assumptions

Amount of Hay (tons)	50
Bale size (ton)	0.5
Avg. Roadside Distance (miles)	0.33
Round trip distance to collection center (miles)	20
Tractor:	
weight (lbs)	9,000
front-to-rear weight distribution	0.33
fuel efficiency (hp-hr/gal)	13
Heating Value of Diesel No. 2 (BTU/gal)	138,110
Heating Value of Ethanol (BTU/gal)	73,000
Rake weight (lbs)	500
Avg. Rake Distance (ft/bale)	500
Tractor-trailer mileage (mpg)	6.2
Pick-up truck mileage (mpg)	10
Ethanol Yield (gal/ton)	80

	MJ	Btu	hp-hr	liter	gallon
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Cutting

Energy Requirements

Implement	295 279,230	110
Tractor	1,231 1,166,775	458

Fuel Requirements

32.08.4

Raking**Energy Requirements**

Front wheels	13 12,711	5
Rear wheels	29 27,733	11
Rake wheels	3 2,825	1
Implement Total	46 43,269	17
Tractor	191 180,802	71

Fuel Requirements

5.01.3

Baling**Energy Requirements**

Implement	268 254,247	100
Tractor	1,121 1,062,385	417

Fuel Requirement

29.17.7

Roadside**Energy Requirement**

Front wheels	36 34,452	14
Rear wheels	125 118,122	46

Moving Total	161 152,574	60
Tractor	672 637,537	250

Fuel Requirement	17.54.6
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Transport

Tractor-trailer (20 bales per trip)

Energy Requirement	2,350 2,227,581	875
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Fuel Requirement	61.1 16.1
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Pick-up truck (6 bales per trip)

Energy Requirement	4,856 4,603,667	1,809
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Fuel Requirement	126.2 33.3
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Total

	MJ BTU	hp-hr	liter	gallon
Tractor-trailer	5,564 5,275,080	2,073	144.6	38.2
Pick-up truck	8,070 7,651,166	3,006	209.7	55.4

Efficiency

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Energy Requirement	MJ/tonne	BTU/ton
(tractor-trailer)	123	105,502

Energy Output	MJ/tonne	BTU/ton
	6,790	5,840,000

Output Fuel Energy : Harvest Energy Requirements	55.4
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APPENDIX A

Definition of Water Quality Constituents

Chemical oxygen demand (COD): The oxygen equivalent of that portion of organic matter in a sample susceptible to oxidation by a strong oxidant. Chemical oxygen demand often is higher than biological oxygen demand (BOD) because more compounds can be chemically oxidized than can be biologically oxidized.

Total solids: The sum of the dissolved and suspended solids in water or wastewater. The residue remaining when water is evaporated away from a sample, and then dried at a specified temperature.

Fixed solids: The portion of total solids remaining as an ash or residue when heated at a specified temperature and time (600 °C for one hour).

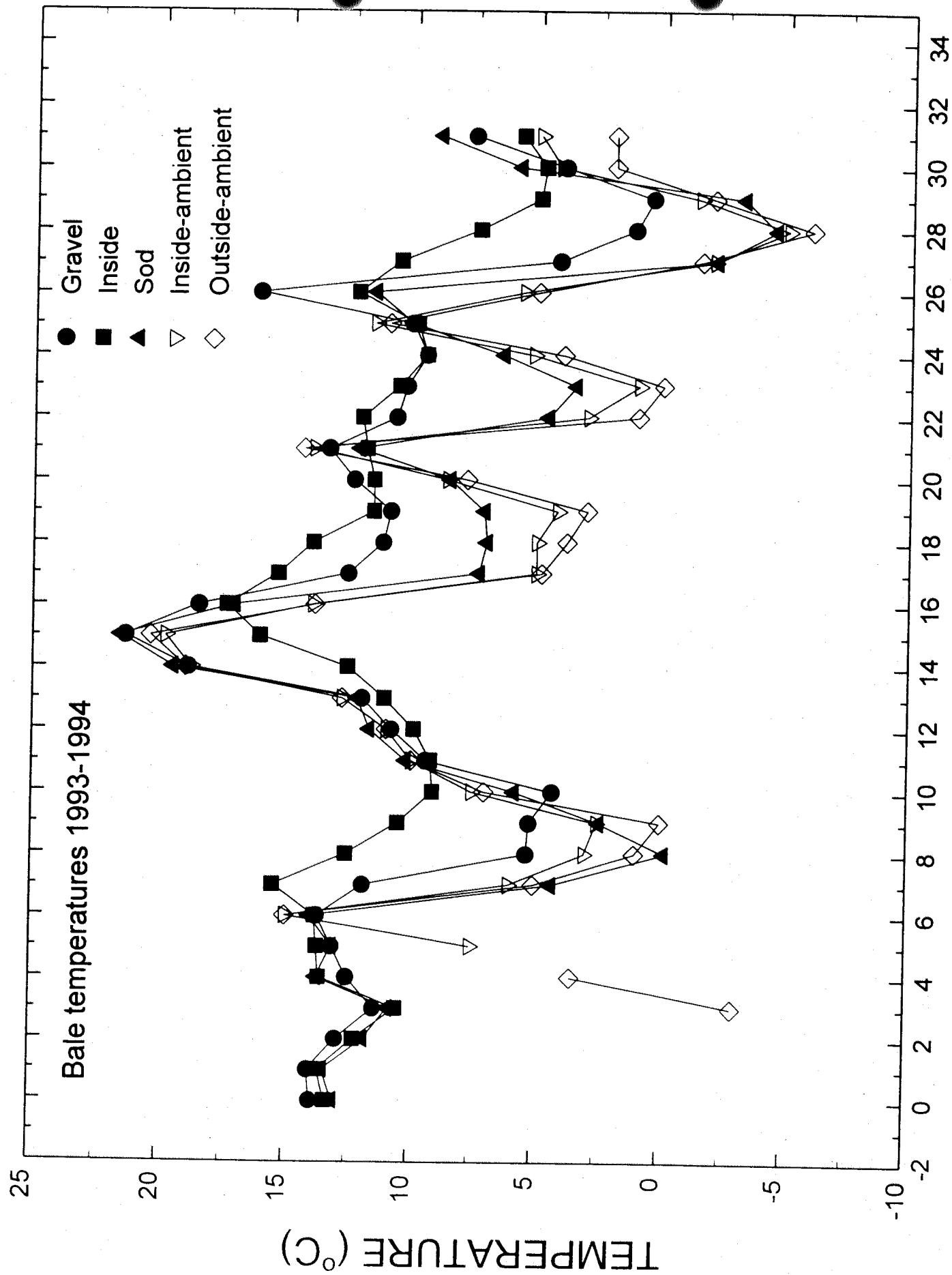
Volatile solids: That portion of the total solids driven off as volatile (combustible) gases at a specified temperature and time (600 °C for one hour).

Total dissolved solids: That portion of total solids retained on a specified filter.

Fixed dissolved solids: The portion of dissolved solids remaining as an ash or residue when heated at a specified temperature and time.

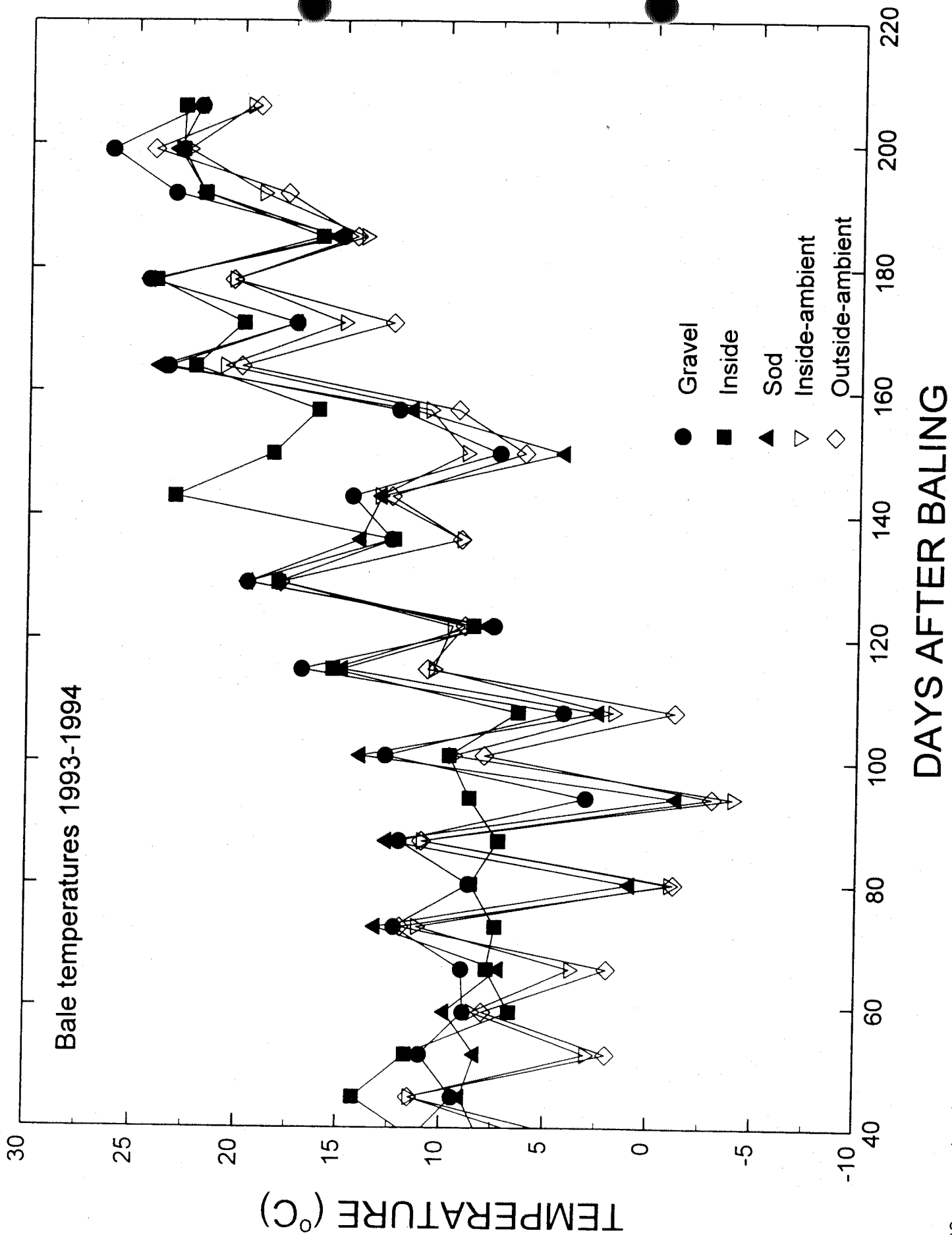
Volatile dissolved solids: That portion of the suspended solids driven off as volatile (combustible) gases at a specified temperature and time (600 °C for at least 20 minutes).

The definitions are taken from Am. Soc. Agric. Eng. Standard ASAE S292.5 Uniform Terminology for Rural Waste Management. p. 462-465. In ASAE Standards, 1992, 39th ed. St. Joseph, MI., and from Metcalf and Eddy, Inc. (1991).



DAYS AFTER BALING

bt2.spw nrel main disk Figure 1. Bale temperatures from 0 to 31 days after baling.



bt3.spw nrel main disk Figure 2. Bale temperatures from 32 to 205 days after baling.

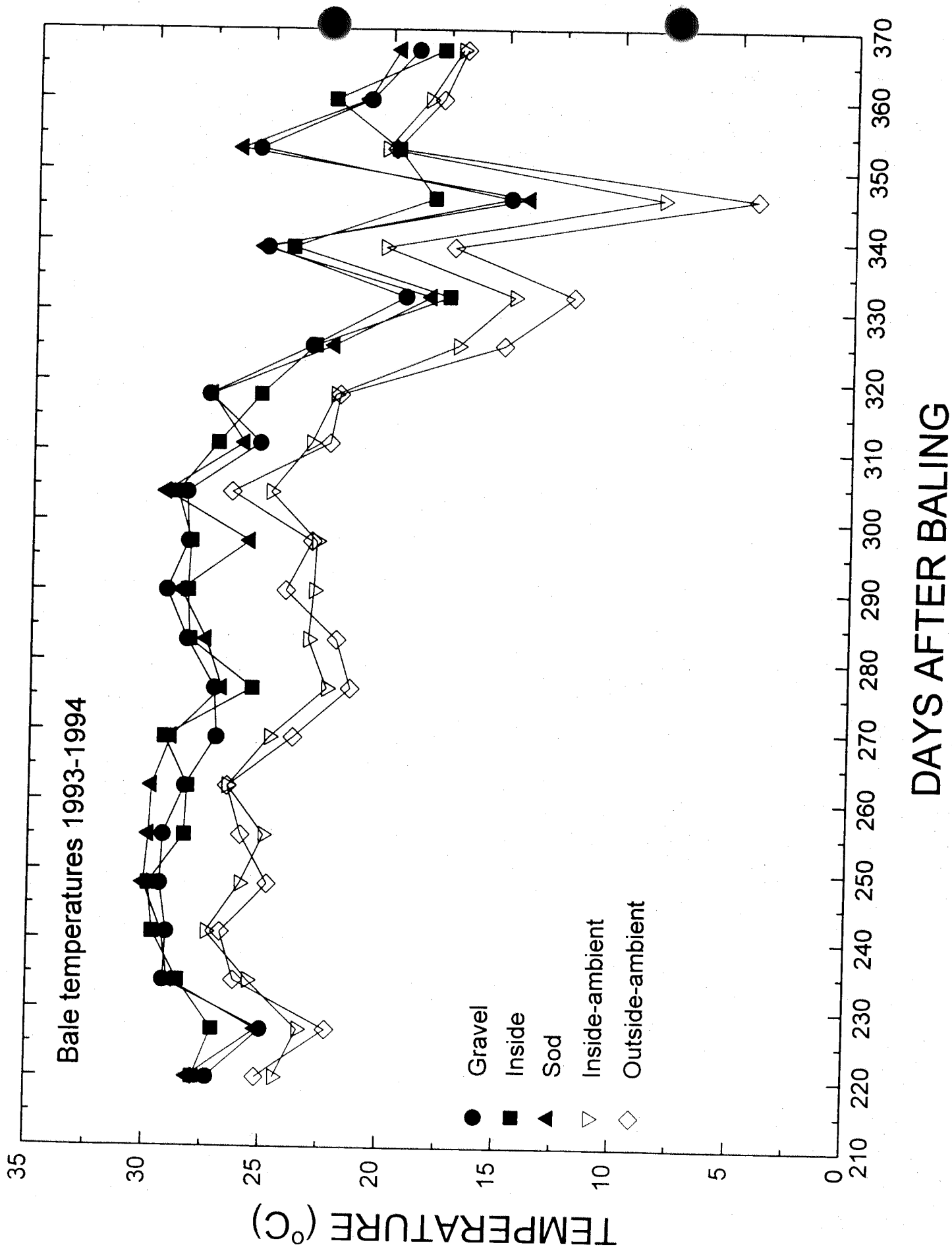


Figure 3. Bale temperatures from 215 to 365 days after baling.

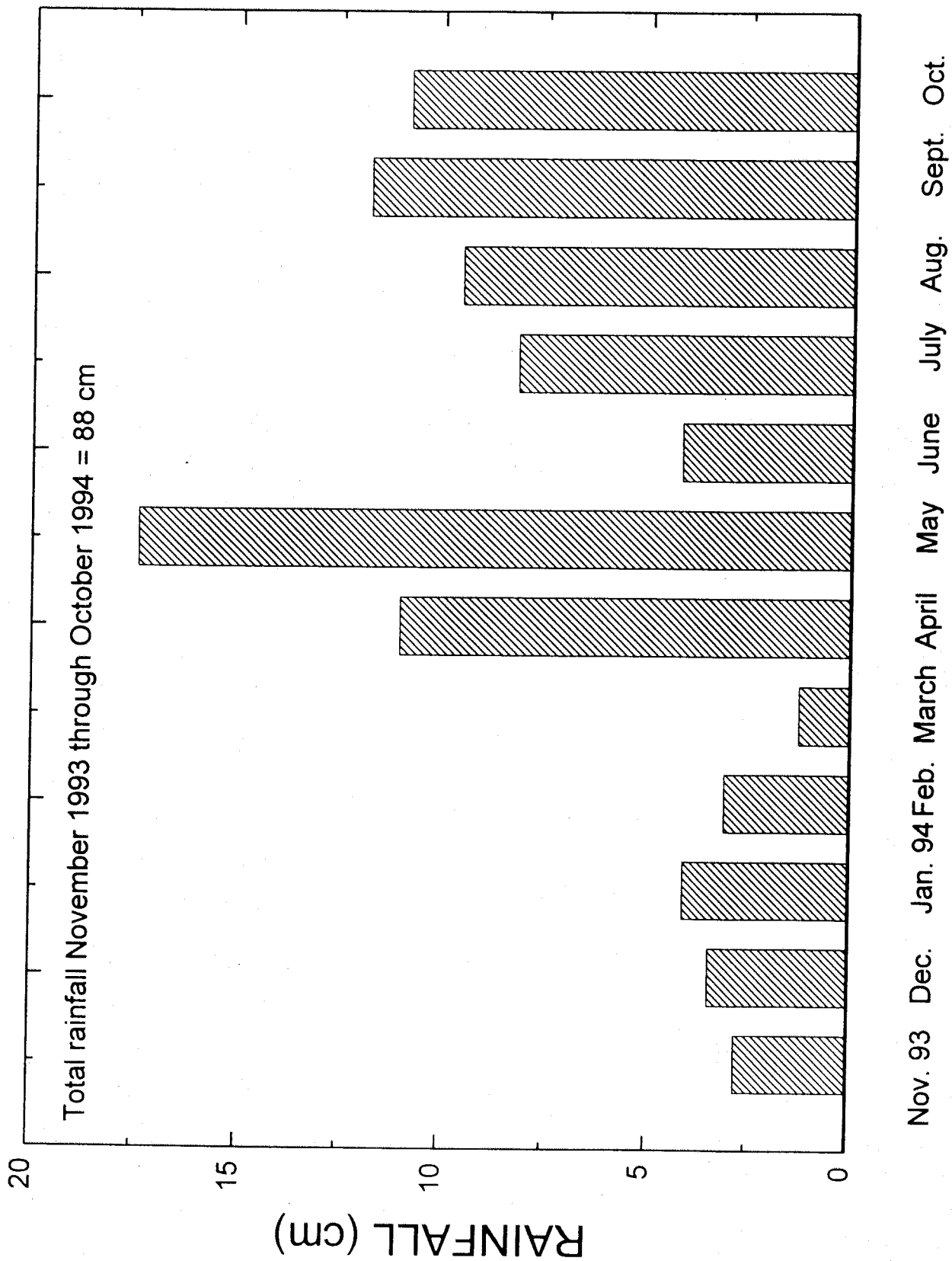


Figure 4. Monthly rainfall for the 12-month storage period.

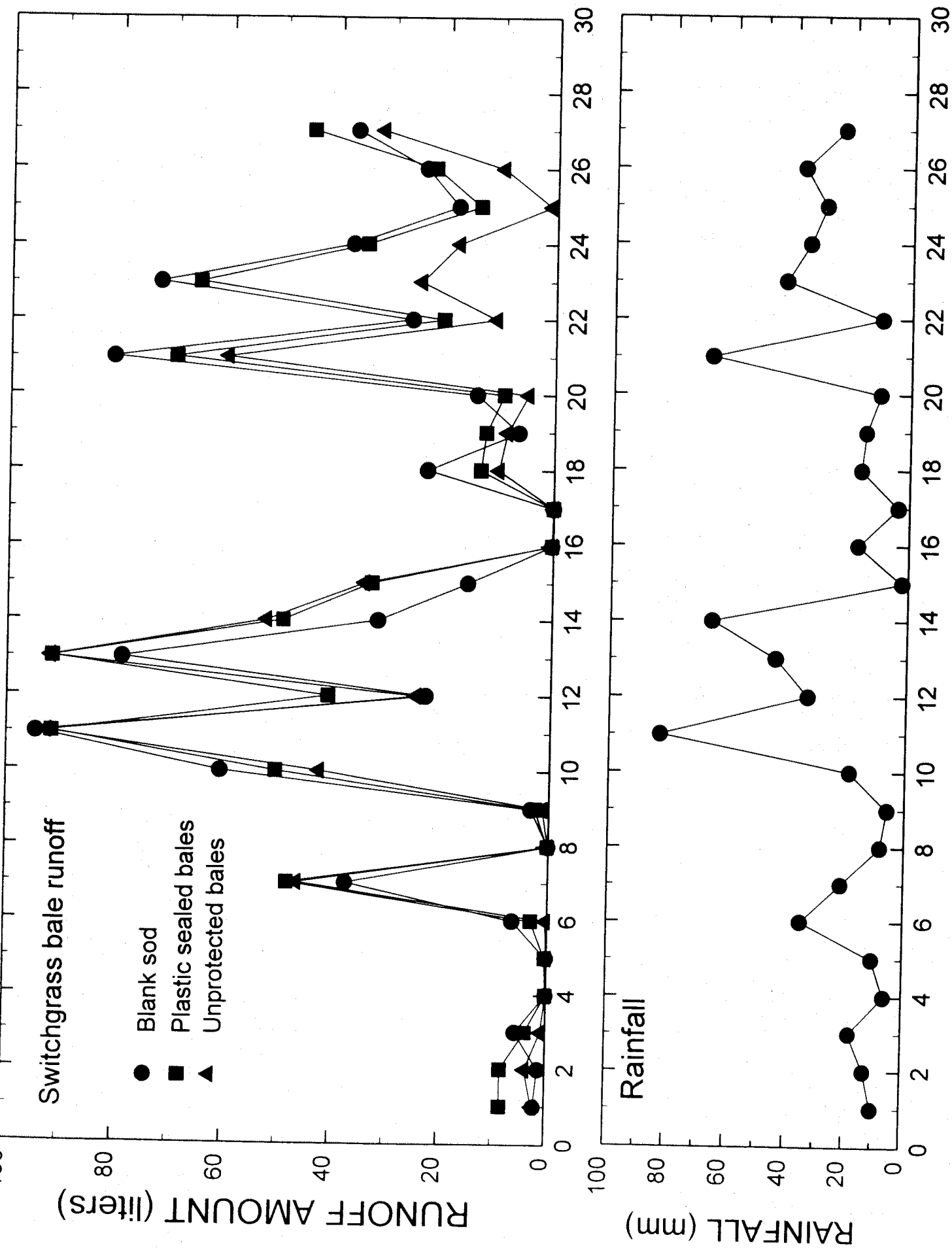


Figure 5. Runoff volume from three treatments during November 1993 to November 1994.

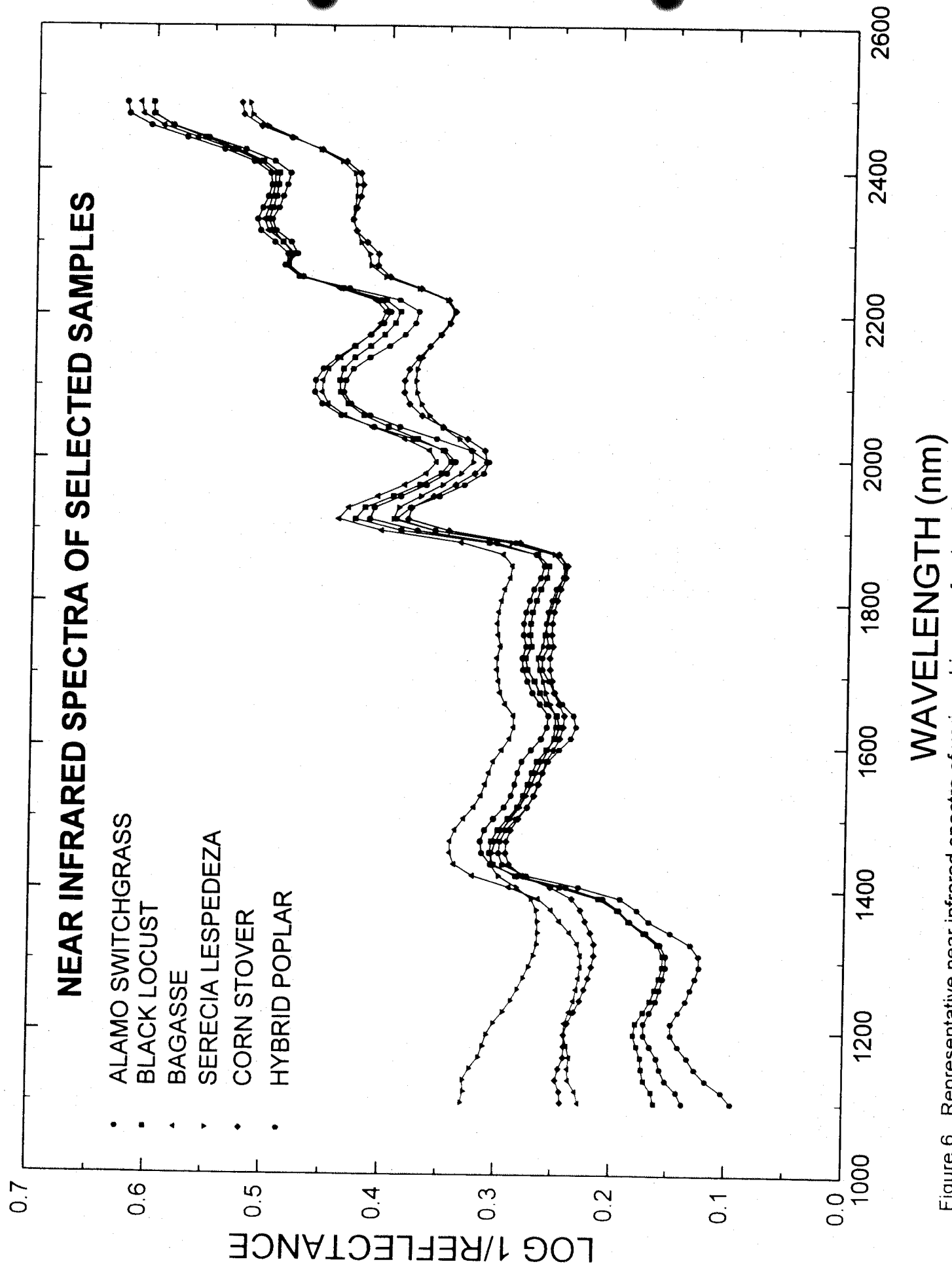


Figure 6. Representative near infrared spectra of various biomass feedstocks

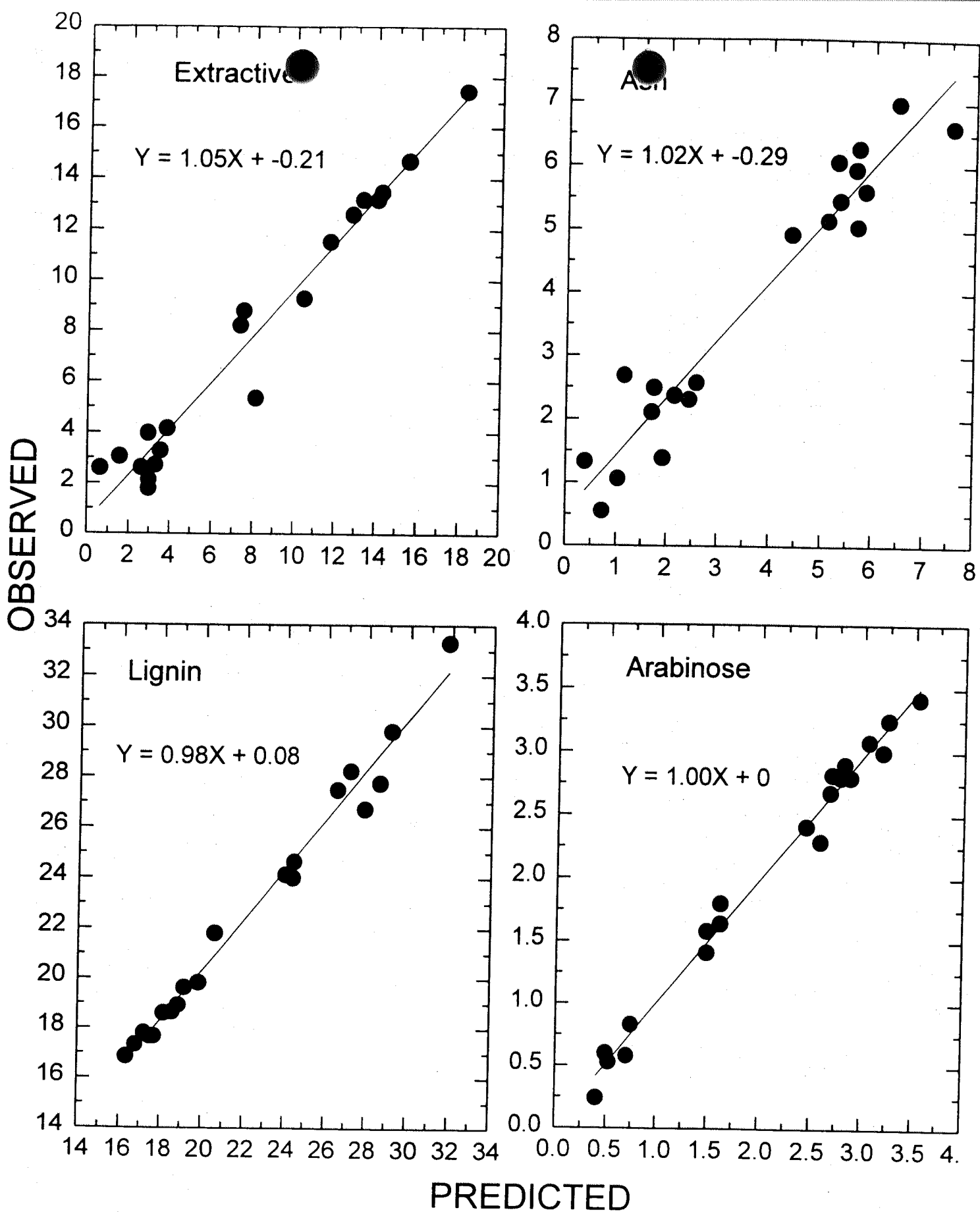
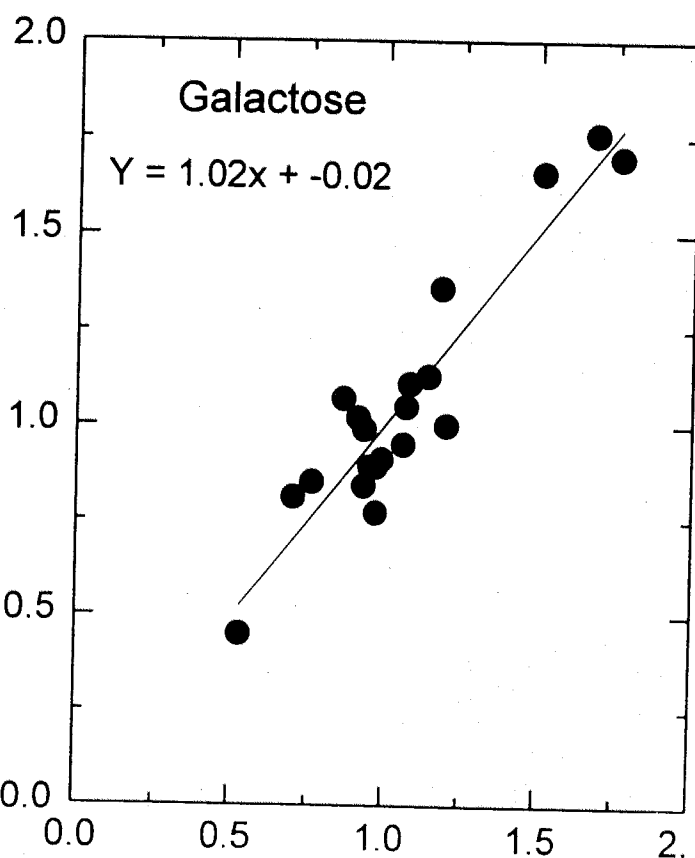
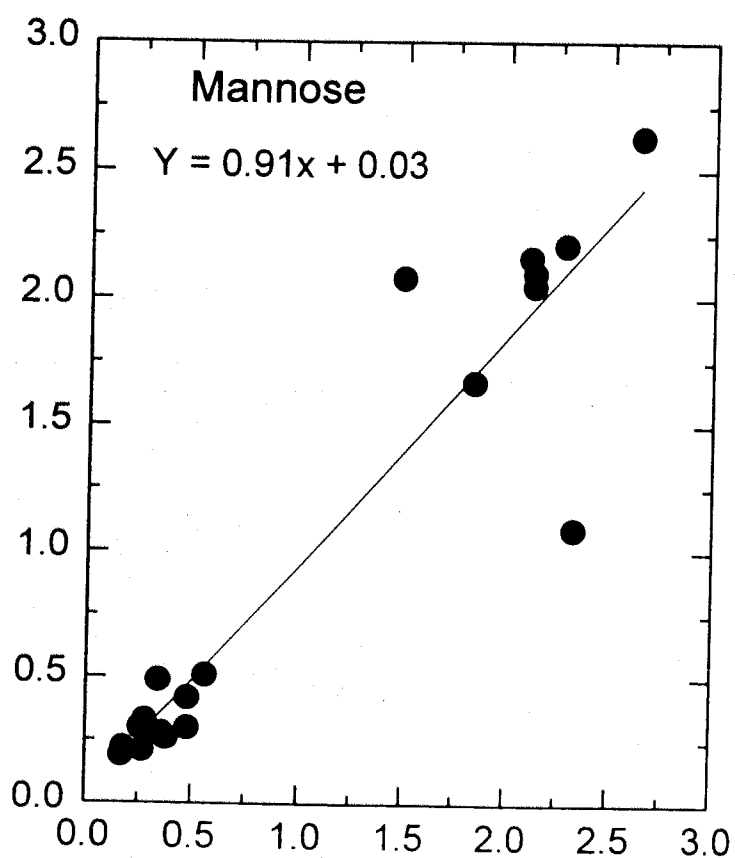
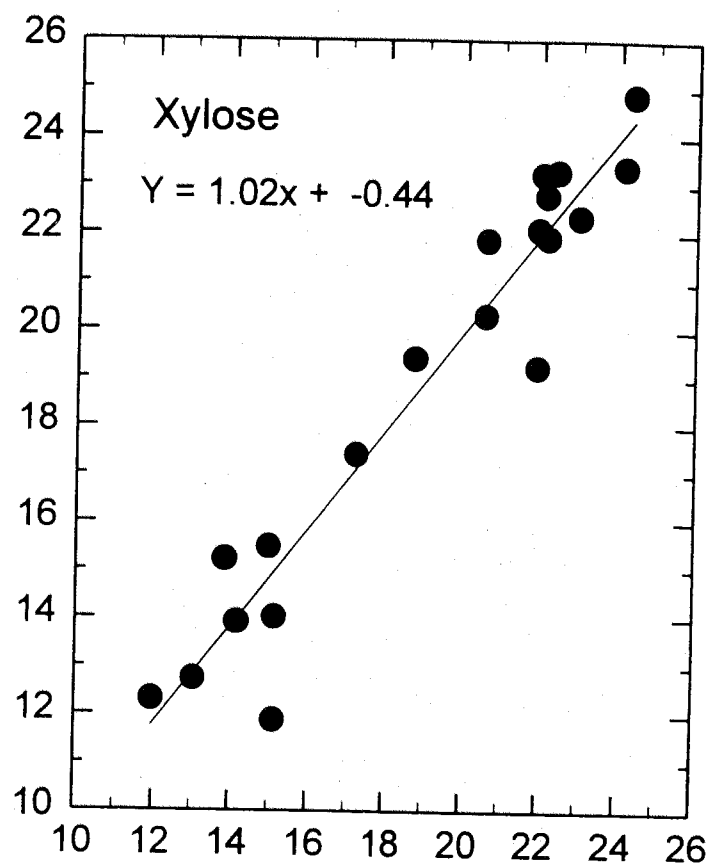
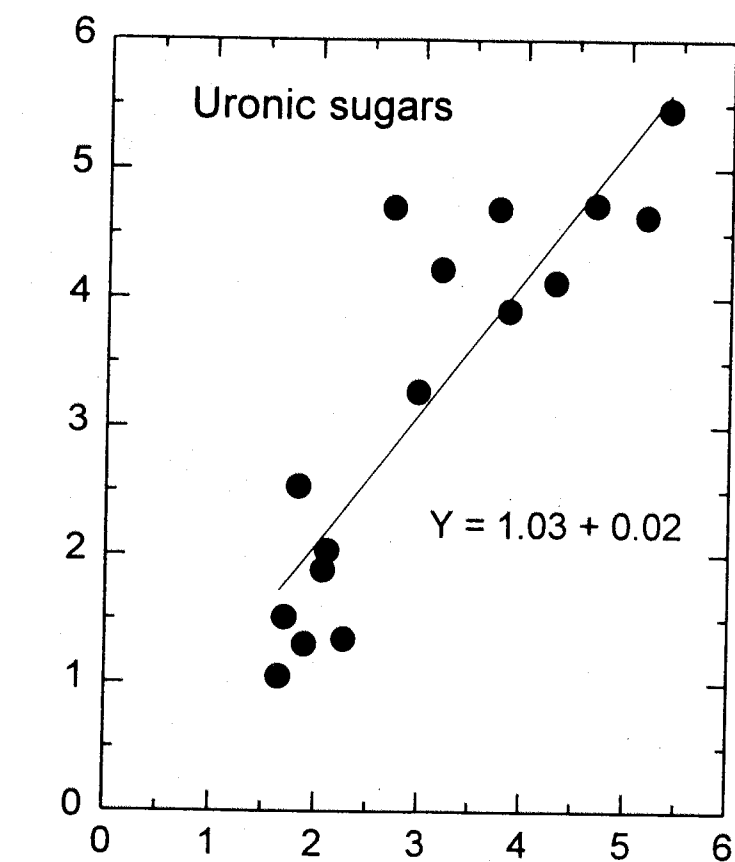


Figure 7. Regression of observed values for extractives, ash, lignin, and arabinose on NIRS predicted values

OBSERVED



PREDICTED

Figure 8. Regression of observed values for uronic sugars, xylose, mannose, and galactose on NIRS predicted values

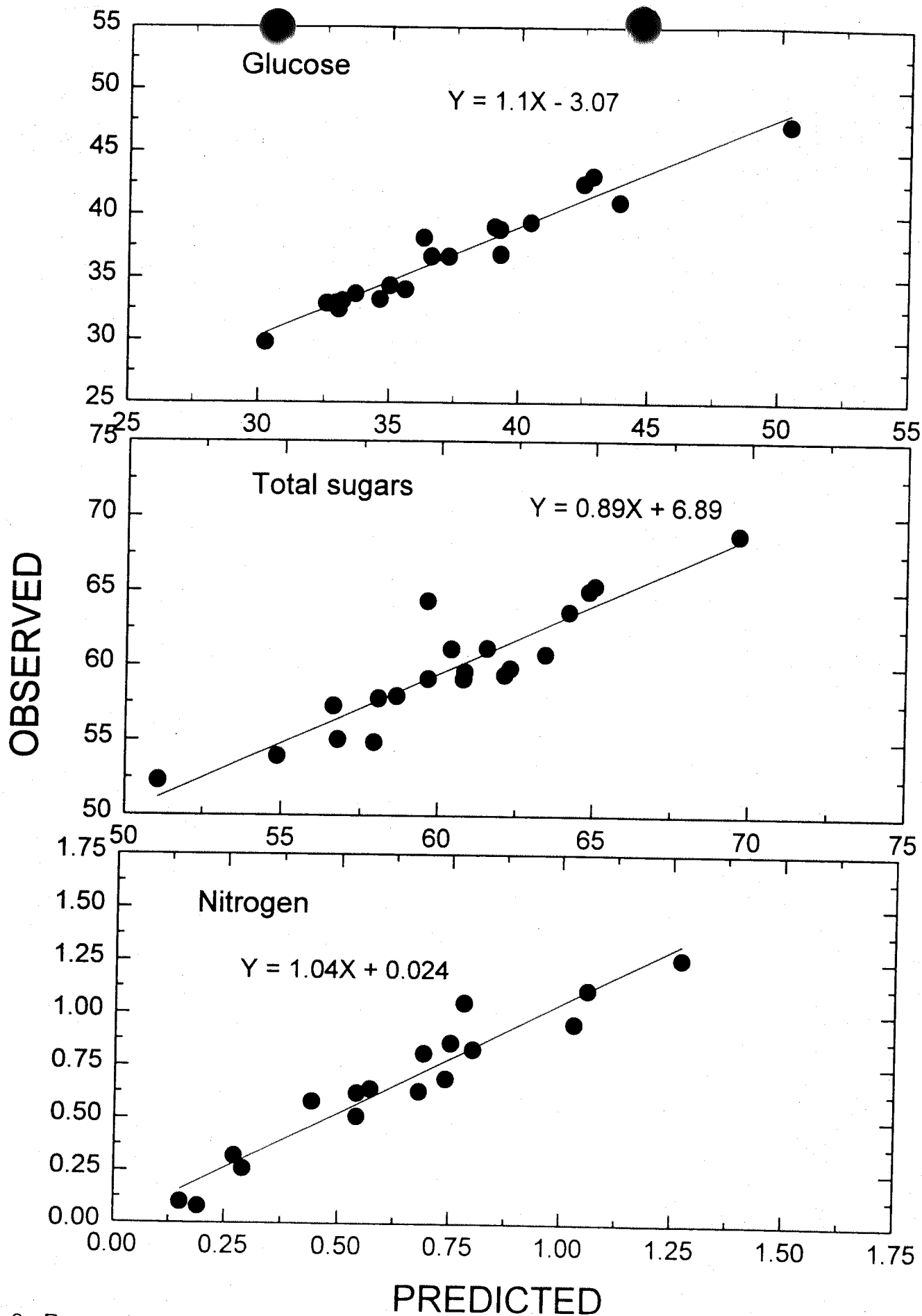


Figure 9. Regression of observed values for glucose, total sugars, and nitrogen on NIRS predicted values
rel 95-3 nrel.nir.spw